Status: Currently Official on 14-Feb-2025
Official Date: Official Prior to 2013
Document Type: USP Monographs
DocId: GUID-FDF9F017-1B6A-4A35-AC87-21FA342BAA85_1_en-US
DOI: https://doi.org/10.31003/USPNF_M26010_01_01
DOI Ref: i0bzq

Digoxin Oral Solution

» Digoxin Oral Solution contains, in each 100 mL, not less than 4.50 mg and not more than 5.25 mg of digoxin ($C_{41}H_{64}O_{14}$).

Packaging and storage—Preserve in tight containers, and avoid exposure to excessive heat.

USP Reference standards ⟨11⟩—

USP Digoxin RS

Identification—

A: The retention time of the major peak in the chromatogram of Oral Solution corresponds to that in the chromatogram of the *Standard preparation,* as obtained in the *Assay*.

B: *Chloramine T–trichloroacetic acid reagent*—Mix 10 mL of a freshly prepared solution of chloramine T (3 in 100) and 40 mL of a 1 in 4 solution of trichloroacetic acid in dehydrated alcohol.

Spotting solvent—Prepare a mixture of chloroform and methanol (2:1).

Standard solution—Dissolve an accurately weighed quantity of USP Digoxin RS in *Spotting solvent* to obtain a solution containing 0.25 mg per mL.

Test solution—Pipet a volume of Oral Solution, equivalent to 0.5 mg of digoxin, into a separator. Add sufficient water to obtain a final volume of approximately 50 mL. Extract the aqueous layer with three 30-mL portions of chloroform, combining the extracts in a conical flask. Evaporate the combined chloroform extracts on a steam bath with the aid of a current of air to dryness. Add 2 mL of *Spotting solvent* to the residue, and shake for 2 minutes.

Procedure—Apply 10 µL of the *Test solution* and 10 µL of the *Standard solution* on a line parallel to and about 2.5 cm from the bottom edge of a reversed-phase thin-layer chromatographic plate coated with a 0.25-mm layer of chromatographic silica gel mixture to which is permanently bonded octadecylsilane (C18). Allow the spots to dry, and place the plates in a developing chamber containing a mixture of methanol and water (7:3). Develop the chromatogram until the solvent front has moved about 15 cm above the line of application. Remove the plate, and allow the solvent to evaporate. Spray the plate with *Chloramine T–trichloroacetic acid reagent*, freshly mixed, and heat in an oven at 110° for 10 minutes. Examine the plate under long-wavelength UV light: the R_F value of the principal spot in the chromatogram of the *Test solution* corresponds to that in the chromatogram of the *Standard solution*.

Alcohol Determination ⟨611⟩: between 90.0% and 115.0% of the labeled amount of C_2H_5OH.

Assay—

Mobile phase—Prepare a filtered and degassed mixture of water, acetonitrile, and isopropyl alcohol (70:27.5:2.5). Make adjustments if necessary (see *System Suitability* under *Chromatography ⟨621⟩*).

Standard preparation—Dissolve an accurately weighed quantity of USP Digoxin RS in diluted alcohol, and dilute quantitatively and stepwise with diluted alcohol to obtain a solution having a known concentration of about 20 µg per mL.

Assay preparation—Transfer an accurately measured volume of 10.0 mL of Oral Solution, equivalent to about 500 µg of digoxin, to a 25-mL volumetric flask, dilute with diluted alcohol to volume, and mix.

System suitability preparation—Prepare as directed in the *Assay* under *Digoxin*.

Chromatographic system (see *Chromatography ⟨621⟩*)—The liquid chromatograph is equipped with a 218-nm detector and a 4.6-mm × 15-cm column that contains packing L1. The flow rate is about 0.5 mL per minute. Chromatograph the *System suitability preparation,* and record the peak responses as directed for *Procedure:* the resolution, *R,* between digoxin and digoxigenin bisdigitoxoside is not less than 2.0; the tailing factor for the analyte peak is not more than 2.0; and the relative standard deviation for replicate injections is not more than 2.0%.

Procedure—Separately inject equal volumes (about 10 µL) of the *Standard preparation* and the *Assay preparation* into the chromatograph, record the chromatograms, and measure the responses for the major peaks. Calculate the quantity, in µg, of digoxin ($C_{41}H_{64}O_{14}$) in each mL of the Oral Solution taken by the formula:

$$2.5C(r_U/r_S)$$

in which *C* is the concentration, in µg per mL, of USP Digoxin RS in the *Standard preparation;* and r_U and r_S are the digoxin peak responses obtained from the *Assay preparation* and the *Standard preparation,* respectively.

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
DIGOXIN ORAL SOLUTION	Nam-Cheol Kim Scientific Liaison	BDSHM2020 Botanical Dietary Supplements and Herbal Medicines

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. PF 31(5)

Current DocID: GUID-FDF9F017-1B6A-4A35-AC87-21FA342BAA85_1_en-US
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